Designation: ~~D7356/D7356M – 13~~ D7356/D7356M – 19

Standard Test Method for Accelerated Acid Etch Weathering of Automotive Clearcoats Using a Xenon-Arc Exposure Device[1]

This standard is issued under the fixed designation D7356/D7356M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers an accelerated exposure test intended to simulate defects in automotive clearcoats caused by acid rain[2] that occur at the Jacksonville, Florida exposure site. Exterior exposures at an acid rain test location in Jacksonville, Florida produce etch defects that range from small pits to 12.7 mm [0.5 in.] in diameter or larger acid-etched spots. The latter type of defect is not produced in other acid-etch tests that only produce pits that are smaller than 6.35 mm [0.25 in.] in diameter.[3]

NOTE 1—Digital images of the acid etch defects produced in outdoor acid-rain exposures and in the accelerated test described in this test method are found in Appendix X1.

1.2 The accelerated test described in this test method uses a xenon-arc light source with daylight filter conforming to the requirements of Practice G155. Specimens are sprayed with a simulated acid rain solution and requires the use of a horizontal, flat specimen array in order to allow the acid rain solution to remain on the test specimens for an extended period of time.

1.3 There is no known ISO equivalent to this test method.

1.4 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other, and values from the two systems shall not be combined.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate ~~safety~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards*:[4]

D1293 Test Methods for pH of Water
D4517 Test Method for Low-Level Total Silica in High-Purity Water by Flameless Atomic Absorption Spectroscopy
G113 Terminology Relating to Natural and Artificial Weathering Tests of Nonmetallic Materials
G147 Practice for Conditioning and Handling of Nonmetallic Materials for Natural and Artificial Weathering Tests
G151 Practice for Exposing Nonmetallic Materials in Accelerated Test Devices that Use Laboratory Light Sources
G155 Practice for Operating Xenon Arc Light Apparatus for Exposure of Non-Metallic Materials

3. Terminology

3.1 *Definitions*—Definitions applicable to this standard can be found in Terminology G113.

3.2 *Definitions of Terms Specific to This Standard:*

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[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

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[2] The acid etch test method is covered by a patent. Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to ASTM International Headquarters. Your comments will receive careful considerations at a meeting of the responsible technical committee,[1] which you may attend.

[3] Brennan, P. J, Marino, M., Boisseau, J. and Campbell, D., Accelerated Acid Etch, Part II: Refined Test Procedure to Reproduce Automotive Acid Etch Provides Improved Lab Practicality and Proven Correlation, FSCT, 2006 FutureCoat Proceedings.

[4] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard**

3.2.1 *acid rain, n*—cloud or rain droplets containing pollutants, such as oxides of sulfur and nitrogen, to make them acidic.

3.2.2 *acid rain spots, n*—spots are produced when the sun evaporates standing water on the vehicle's surface.

3.2.2.1 *Discussion*—

They fall into two categories, mineral deposits and acid-etched spots. Mineral deposits are white calcium spots or salt deposits left on the surface after water evaporates. These deposits can be easily removed with a pre-wax cleaner (polish) or a clay bar. Acid-etched spots are depressions in the paint or clear coat, which cannot be easily removed.

3.2.3 *trained assessor, n*—an individual knowledgeable and proficient in the procedure capable of making consistent evaluations based on defined criteria so as to produce a repeatable result.

3.2.3.1 *Discussion*—

In this standard, assessor discerns the difference between water spotting and etching, and judges the magnitude.

4. Summary of Test Method

4.1 The Automotive Clearcoat coating being evaluated is applied to an automotive basecoat (typically black) that has been coated onto a steel panel. All edges of the panel are sealed to prevent corrosion. Coatings applied to other types of panels may also be used.

4.2 Test specimens are placed in a xenon-arc device equipped with a horizontal exposure rack and are exposed to alternating periods of light, spray with acid rain solution, and spray with water. The evaporation of the water from the acid solution on the test specimen surface results in deposits with high acid concentration.

4.3 After 400 ~~hours~~hr of exposure, test specimens are evaluated and rated for etch.

5. Significance and Use

5.1 Acid etch damage is an important warranty claim item for automotive companies. As a result, acid etch resistance is an important parameter for automotive exterior coatings. The method described in this test method has been shown to simulate acid etch damage of automotive clearcoats that occurs when such coatings are exposed from May through mid-August in Jacksonville, FL.[3,5] The accelerated test described in this standard allows year-round testing as opposed to the limited outdoor exposure time available for the Jacksonville, FL exposures.

6. Apparatus

6.1 Xenon-arc exposure device conforming to Practice G151 and Practice G155 with the following additional requirements.

6.1.1 Specimen rack that is within ~~±3 degrees~~ ±3° of horizontal.

6.1.2 Means to spray pure deionized water and acid rain solutions independently, and without cross contamination, from one another.

NOTE 2—Use of dual spray systems, one for acid solution and the other for high purity water has been found to be effective for eliminating cross contamination.

6.1.2.1 The volume of spray water shall be sufficient to flood (with excess) the surface of the specimen within the one-minute spray time. A spray delivery rate of 3 ~~litres~~L/min/m^2 ~~per minute per square metre~~ has been found suitable.

6.1.3 Use of a single spray system has not been evaluated. If a single spray system is used, the following shall be included with the test report:

6.1.3.1 If a single spray system is used for both the acid solution and high purity water, data verifying that there is no cross contamination between the acid spray and the high purity water spray.

6.1.4 Xenon-arc apparatus shall be equipped with daylight filters that meet the requirements of Practice G155.

6.1.5 Xenon-arc apparatus shall be able to simultaneously and automatically control irradiance, black panel temperature, relative humidity and chamber air temperature.

6.2 Xenon-arc apparatus shall be equipped with an uninsulated black panel complying with Practice G151.

6.3 pH meter capable of measuring to ±0.2 units.

7. Reagents and Materials

7.1 *0.02 N H_2SO_4 Sulfuric Acid Volumetric Solution,* (accurate to ±1 part per 1000).

7.2 *0.3 % HNO_3 (Nitric Acid),* by weight or v/v.

[5] Boisseau, J. and Pattison, L., BASF Corporation, and Henderson, K. and Hunt, R., Bayer Material Science, "The Flaws in Accelerated Weathering of Automotive OEM Coatings," Paint and Coatings Industry, June 2006.

7.3 *0.02 N NaOH Sodium Hydroxide Volumetric Solution,* (accurate to ± 1 part per 1000).

7.4 *0.01 M $CaCl_2$,* (Calcium Chloride Standard for water hardness testing).

7.5 *0.01 M KCl (Potassium Chloride) Conductivity Solution,* calibration standard solution 1413 ~~micro-ohms.~~μΩ.

8. Apparatus Setup

8.1 Operate, maintain and calibrate the apparatus to manufacturer's specifications. ~~The test should not be interrupted once it has started (excluding daily panel reposition). Calibrations and maintenance should be completed~~ To the extent possible, perform all calibrations and maintenance before the test starts or after the test has ~~completed.~~been completed. If the test is interrupted for any reason (excluding daily specimen repositioning), it may affect test results and as such, the reason for the interruption and its duration shall be noted on the test report.

8.2 Water used for spray and humidification shall have a maximum of 1 ppm total solids and a maximum of 0.2 ppm silica. Unless otherwise specified, determine silica levels in accordance with Practice D4517. A combination of deionization and reverse osmosis treatment can effectively produce water with the desired purity.

8.3 *Acid Rain Solution*—The composition for the acid rain solution is as follows:

8.3.1 Add 977.5 g of deionized water to a 1 ~~litre~~L flask and then add the following:

15.0 ~~grams~~g – 0.02 N H_2SO_4 (Sulphuric Acid)
2.2 ~~grams~~g – 0.3 % HNO_3 (Nitric Acid)
2.5 ~~grams~~g – 0.02 N NaOH (Sodium Hydroxide)
1.8 ~~grams~~g – 0.01 M $CaCl_2$ (Calcium Chloride)
1.0 ~~grams~~g – 0.01 M KCl (Potassium Chloride)

8.3.2 This provides a 1000 g ~~(1 L)~~[1 L] solution with a pH of 3.3 to 3.5, which is similar to that of Jacksonville rain water.

TABLE 1 Accelerated Acid Etch Exposure Cycle Sequence

Accelerated Acid Test Exposure Cycle		
Step 1	1 min	Dark exposure; with acid rain spray
Step 2	3 hr 50 min	Dark exposure; 40°C uninsulated black panel (B.P.) temperature (monitored not controlled); 40°C chamber air temperature; 80 % RH
Step 3	12 hr	Light exposure; 0.55 W/m²/nm at 340 nm; daylight filters; 65°C uninsulated B.P.; 50°C chamber air temperature; 80 % RH
Step 4	27 min	Dark exposure; 40°C uninsulated B.P. temperature (monitored not controlled); 40°C chamber air temperature; 80 % RH
Step 5	1 min	Dark exposure; with pure water (DI) spray
Step 6	3 hr 50 min	Dark exposure; 40°C uninsulated B.P. temperature (monitored not controlled); 40°C chamber air temperature; 80 % RH
Step 7	1 min	Dark exposure; with pure water (DI) spray
Step 8	3 hr 50 min	Dark exposure; 40°C uninsulated B.P. temperature (monitored not controlled); 40°C chamber air temperature; 80 % RH

~~Note—Chamber~~Note—Chamber air temperature and black panel temperature are controlled during the light period. Chamber air is controlled during the dark steps and the uninsulated black panel temperature is monitored. The % RH is controlled during light and dark periods where specified.

8.3.3 After the water has cooled to room temperature and stirred for 24 ~~hours,~~hr, measure the pH of the solution with a pH meter following Practice D1293. If the solution pH is not between 3.3 and 3.5 make a new solution.

8.4 Expose the test specimens to the cycle shown in Table 1.

8.4.1 Refer to ~~Table X3.2,~~Table X3.2, Operational Fluctuations on Exposure Conditions, in Practice G155 for the maximum allowed fluctuations about the set points. If the operational fluctuations are greater than the maximum allowed after the equipment has stabilized, discontinue the test and correct the cause of the problem before continuing.

9. Test Procedure

9.1 Apply coating to be evaluated to the desired substrate. After coating is applied and dried or cured, seal any cut edges of the test specimens. An air-dry primer or silicone sealant has been found to be suitable for sealing cut edges. The exposed surface of all test specimens must be clean and free from finger prints or other surface contaminants. All other procedures for specimen handling and identification shall be in accordance with Practice G147.

9.2 Place specimens in the xenon-arc exposure device and program the device to run the exposure cycle described in Table 1, starting at Step 1 (dark exposure with acid spray). Unless otherwise specified, set the device timer to provide a 400 ~~hour~~hr exposure. Refer to the device manufacturer's instructions for proper operation.

9.2.1 Once the exposure has started, do not interrupt the exposure more than once per day. The single test interruption shall be for specimen repositioning and placement or removal. Additional interruptions may increase variability of test results.

9.3 The volume of water and acid spray for the test must be excessive, running off the panels. A spray volume of at least ~~3~~3 L/min/m² ~~litres per min per square metre~~ has been found acceptable.

9.4 Periodic repositioning of specimens during the exposure period is required to ensure that each receives an equal amount of exposure to the acid solution and pure water spray. Reposition the panels daily during the light cycle. Follow the manufacturers guidelines for the repositioning pattern. See Fig. 1 for an example of how samples are repositioned. Repositioning of specimens on the weekends is not required.

9.5 Report the acid damage as agreed upon by contractual parties.

10. Evaluation Report

10.1 The evaluation of acid damage is performed visually and the test specimen is rated on a scale from 0 (best) to 10 (worst). A summary of the rating scale is shown in Table 2 ~~and Table 3~~. Other evaluations can be used if agreed ~~up~~to by parties concerned.

TABLE ~~3~~ Detailed Acid Etch Ratings

Rating	Description	Skill Level
~~0~~	~~No damage~~	~~N/A~~
~~1~~	~~Slight pitting damage, no acid etch spots~~	~~trained observer~~
~~2~~	~~Moderate pitting – no acid etch spots~~	~~trained observer~~
~~3~~	~~Few full or partial acid etch spots start~~	~~trained observer~~
~~4~~	~~Full acid etch spots start to appear (light)~~	~~untrained observer~~
~~5~~	~~Full acid etch spots, inc. frequency, few deeper spots~~	~~untrained observer~~
~~6~~	~~Inc. frequency, inc. depth, borderline paint repair~~	~~untrained observer~~
~~7~~	~~Frequency and depth would require repaint~~	~~all observers~~
~~8~~	~~Start to see acid etch spots overlap previous spots~~	~~all observers~~
~~9~~	~~Inc. frequency, depth of spots, overlap~~	~~all observers~~
~~10~~	~~High % area damaged, spots overlap, discoloration~~	~~all observers~~

TABLE 2 Detailed Acid Etch Ratings

Rating	Description	Skill Level
0	No damage	trained assessor
1	Slight pitting damage, no acid etch spots	trained assessor
2	Moderate pitting – no acid etch spots	trained assessor
3	Few full or partial acid etch spots start	trained assessor
4	Full acid etch spots start to appear (light)	untrained assessor
5	Full acid etch spots, inc. frequency, few deeper spots	untrained assessor
6	Inc. frequency, inc. depth, borderline paint repair	untrained assessor
7	Frequency and depth would require repaint	any assessor
8	Start to see acid etch spots overlap previous spots	any assessor
9	Inc. frequency, depth of spots, overlap	any assessor
10	High % area damaged, spots overlap, discoloration	any assessor

Note—Comments on ratings: Rating 0 to 3 - The etch spots are very faint and only noticed by a trained assessor. Rating 4 to 6 - The etch spots are slight to moderate and in some cases noted by an untrained assessor (for example, vehicle owner). Rating 7 to 10 - The etch spots are severe enough that any assessor would notice.

Example of pictorial ratings are shown in Appendix X2.

11. Exposure Report

11.1 The exposure report is as agreed upon by contractual parties.

11.2 The Exposure Control/Report form shall include the following additional information:

11.2.1 Laboratory ~~Name.~~name.

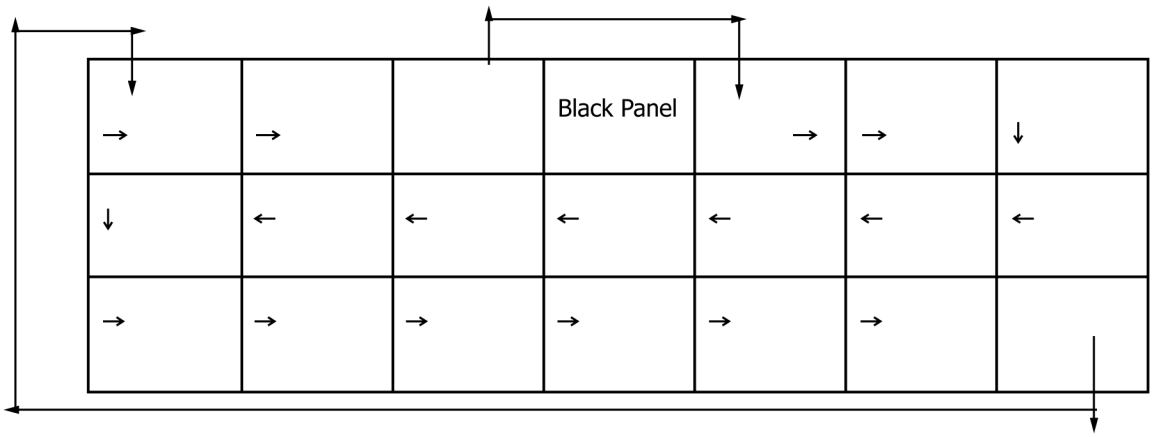

FIG. 1 Example of Repositioning Pattern

11.2.2 Type and serial number of exposure equipment.
11.2.3 Month and ~~Year~~year of equipment operation.
11.2.4 Test method.
11.2.5 Frequency of operation verification, for example, three or seven day intervals.
11.2.6 Daily record of black panel temperature (BPT).
11.2.7 Daily record of chamber temperature.
11.2.8 Daily record of irradiance.
11.2.9 Test interruptions (if any), duration and reasons.

12. Precision and Bias

12.1 The test results are a visual comparison to a pictorial standard and the ranking of the test sets. The repeatability standard deviation (Table ~~4~~3) has been determined to be:

TABLE 43 Repeatability for Lab A

Coating	Set #1	Set #2	Set #3	Set #5	Mean	Standard Deviation
Paint A	3	5	4	6	4.5	1.29
Paint B	6	6	6	7	6.25	0.50
Paint C	7	9	9	8	8.25	0.96
Paint D	10	10	10	10	10	0.00

12.1.1 The Spearman rank correlation coefficient was 1.0 for all possible comparisons between the different sets. The Pearson linear correlation coefficients for comparisons of the data in the different sets were as follows:

Set 1 to Set 2 = 0.92
Set 1 to Set 3 = 0.93
Set 1 to Set 4 = 0.98
Set 2 to Set 3 = 0.99
Set 2 to Set 4 = 0.94

12.2 The reproducibility of this test method cannot be determined at this time because of the lack of volunteers for round robin testing.

13. Keywords

13.1 accelerated weathering; acid etch; acid rain; automotive; clearcoats; Jacksonville; xenon-arc

APPENDIXES

(Nonmandatory Information)

X1. PHOTOS OF ACCELERATED WEATHERING XENON ACID ETCH TEST RESULTS FOR AUTOMOTIVE CLEARCOATS VS. FIELD EXPOSURES

X1.1 Acid etch images. See Fig. X1.1, Fig. X1.2, Fig. X1.3, and Fig. X1.4.

FIG. X1.1 Digital Photograph of Acid Etch Produced During Field Exposure (Jacksonville, FL, ~~50 x~~ 50× magnification)

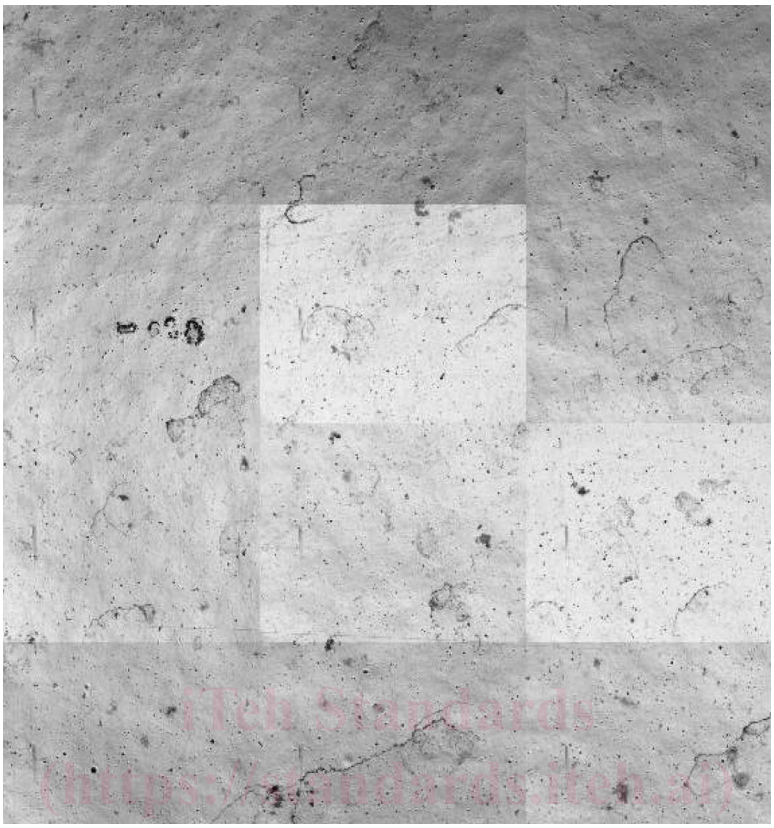

FIG. X1.2 Digital Photograph of Acid Etch Produced During Field Exposure (Jacksonville, FL, actual size 10 × ~~10 in.~~)10 in. [25.40 × 25.40 cm])